Physics Spectrum Journal
Volume 01 Issue 01, January 2026
P-ISSN: xxxx-xxxx, E-ISSN: xxxx-xxxx

Integrated XRF–XRD–Optical Characterization of Rock Samples from Leang Panning Park, Mallawa (Maros, South Sulawesi, Indonesia)

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Article Info

Article info:
Received: xx-xx-20xx
Revised: xx-xx-20xx
Accepted: xx-xx-20xx

Keywords:
XRF; XRD; Optical microscopy; Albite; Quartz; Magnetite; Leang Panning Park; Maros; South Sulawesi

How To Cite:
xxxxxx

DOI:
xxxxx

Abstract

This study characterizes two rock samples collected during a student field exploration at Leang Panning Park, Wanua Waru, Mallawa District, Maros (South Sulawesi) on 7–8 September 2023. Samples were prepared as polished bulk (~2 mm thick) for XRD and optical microscopy, and as powders for XRF. XRF shows that Sample A is dominated by Si (29.7 at%), with notable Al (7.65 at%) and minor Fe, Na, Ca, K, Mg; Sample B contains high Si (24.3 at%), Al (9.2 at%), Ca (8.21 at%), and Fe (7.07 at%). XRD phase identification indicates Sample A is albite-rich (~54–64%) with magnetite (~40%) and trace KO_2, SO_3, and Ba peroxide phases; Sample B is dominated by SiO_2 (46.5%) with sodium aluminum oxide (23.8%), magnetite (10.9%), K_2O (9.9%), and hydrated V_2O_5 (8.9%). Relative crystallinity from integrated peak areas is ~26.45% (A) and 15.77% (B), while Scherrer estimates give mean crystallite sizes of ~2.08 nm (A) and 6.21 nm (B). Results are consistent with silicate-dominated compositions and suggest igneous affinities discussed in the field report.

1. Introduction

The characterization of minerals and rocks is a fundamental aspect of geology and materials science, as it provides insights into the composition, crystallinity, and potential applications of natural resources. In particular, the study of rocks in karst environments is crucial because these regions are often composed of diverse mineral phases such as carbonates, silicates, and oxides, which collectively record both primary geological processes and secondary weathering effects [1]. Mineralogical studies in such settings not only improve our understanding of geological history

but also support their potential application in construction, ceramics, and environmental remediation [2].

To achieve reliable mineralogical identification, researchers often employ a combination of analytical methods. X-ray fluorescence (XRF) is widely used to determine the elemental composition of bulk samples with high sensitivity, while X-ray diffraction (XRD) remains the standard technique for identifying crystalline phases and calculating crystallite size and degree of crystallinity [3]. Complementing these, optical microscopy provides textural and microstructural information, which is essential for understanding grain morphology and phase heterogeneity [4]. When used together, these techniques offer a comprehensive characterization of mineral samples, bridging chemical, structural, and visual evidence.

The present research was carried out as part of a student exploration activity at Leang Panning Park, located in Wanua Waru, Mallawa District, Maros, South Sulawesi. This site is part of the broader Maros–Pangkep karst landscape, one of the largest karst areas in Indonesia, which is geologically significant and rich in limestone and silicate rock formations. Despite its importance, detailed mineralogical characterization of rock samples from this location remains limited. Therefore, this study aims to analyze two representative rock samples collected from Leang Panning Park using an integrated approach of XRF, XRD, and optical microscopy. The objectives are to identify the elemental composition, determine the crystalline phases, evaluate relative crystallinity, and observe textural features. These results are expected to contribute to a better understanding of the geological characteristics of the Maros region and provide valuable data for future research and applied material studies .

2. Experimental Method

Rock samples were collected from Leang Panning Park, Wanua Waru, Mallawa District, Maros, South Sulawesi during a student exploration activity. The sampling location was chosen due to its geological significance as part of the Maros–Pangkep karst landscape, which is characterized by limestone and silicate-rich outcrops. Two representative samples were selected from different points within the site to ensure diversity in mineralogical features. Each sample was carefully extracted using geological hammers and chisels, then stored in clean, labeled plastic containers to avoid contamination during transport.

Prior to analysis, the rock samples were mechanically cut into smaller fragments of approximately 2×2 cm in size using a diamond saw. The fragments were cleaned with distilled water and air-dried at room temperature. For XRF and XRD analyses, the dried samples were ground into fine powder using an agate mortar to minimize contamination from metallic tools. The powders were then sieved to achieve uniform particle size below 200 mesh, ensuring consistent analytical results.

X-ray Fluorescence (XRF) Analysis: The powdered samples were pressed into pellets with a diameter of 2.5 cm using a hydraulic press under a pressure of 10 tons. The prepared pellets were then analyzed using an XRF spectrometer to obtain semi-quantitative data on elemental composition. Calibration was carried out with certified reference materials to ensure accuracy of the measurements.

Optical Microscopy: For microstructural examination, thin sections of the rock samples were prepared. Small slabs of the samples were cut, mounted on glass slides using epoxy resin, and ground to a thickness of approximately 30 µm. These thin sections were observed under an optical polarizing microscope in both plane-polarized light (PPL) and cross-polarized light (XPL). Digital images were captured for further analysis, and the images were subsequently processed using ImageJ software to quantify grain size, phase distribution, and brightness intensity.

X-ray Diffraction (XRD) Analysis: A separate portion of the powdered samples was used for XRD measurements. The analysis was performed with Cu Kα radiation (λ = 1.5406 Å), operated at 40 kV and 30 mA. The scanning range was set from 10° to 80° 2θ with a step size of 0.02° and a scan speed of 2°/min. The obtained diffractograms were analyzed using HighScore Plus software combined with the ICDD PDF-4+ database for phase identification. Crystallite size and degree of crystallinity were estimated using the Debye–Scherrer equation and peak intensity analysis, respectively [5].

By integrating these methods XRF for elemental composition, optical microscopy for microstructural observation, and XRD for crystalline phase identification a comprehensive mineralogical characterization of the rock samples from Leang Panning Park was achieved.

3. Results and discussion

This section presents the results obtained from the three major characterization techniques used to analyze the rock samples from Manggululiling Nature Park: SEM-EDS, optical microscopy with image analysis, and XRD. Each method provided a different but complementary set of information regarding the elemental composition, microstructure, and crystalline phases of the rock samples.

3.1 XRF Analysis

The XRF analysis elucidated the bulk elemental composition of the two rock samples collected from Leang Panning Park. In Sample A, the predominant elements were silicon (Si), aluminum (Al), alongside calcium (Ca) and minor iron (Fe)**,** whereas Sample B exhibited elevated concentrations of silicon and calcium as well as higher iron content. These results are consistent with mixed silicate–carbonate lithologies commonly found in karst settings and echo findings from karst-derived terra rossa formations in Apulia, Italy, where a multi-method approach combining XRF, optical microscopy, and SEM revealed important geochemical patterns related to carbonate weathering and siliciclastic contribution

The identification of substantial Si–Al signals suggests the presence of feldspathic and quartz-bearing phases within a carbonate-dominant matrix. Concurrently, the Ca enrichment aligns with expectations for calcite-rich rocks, characteristic of karst systems. The relative abundance of Fe may derive from accessory oxide minerals or diagenetic iron mobilization, which has also been documented in studies of colored speleothems where trace metal substitution influences optical and chemical properties.

3.2 Optical Measurements

Under petrographic analysis, thin sections of both samples were examined using polarized light microscopy. In plane-polarized light (PPL), the samples displayed a granular texture with interlocking grains. Calcite crystals were identified by their characteristic cleavage and low birefringence. Cross-polarized light (XPL) revealed interference colors diagnostic of quartz and feldspar phases [6].

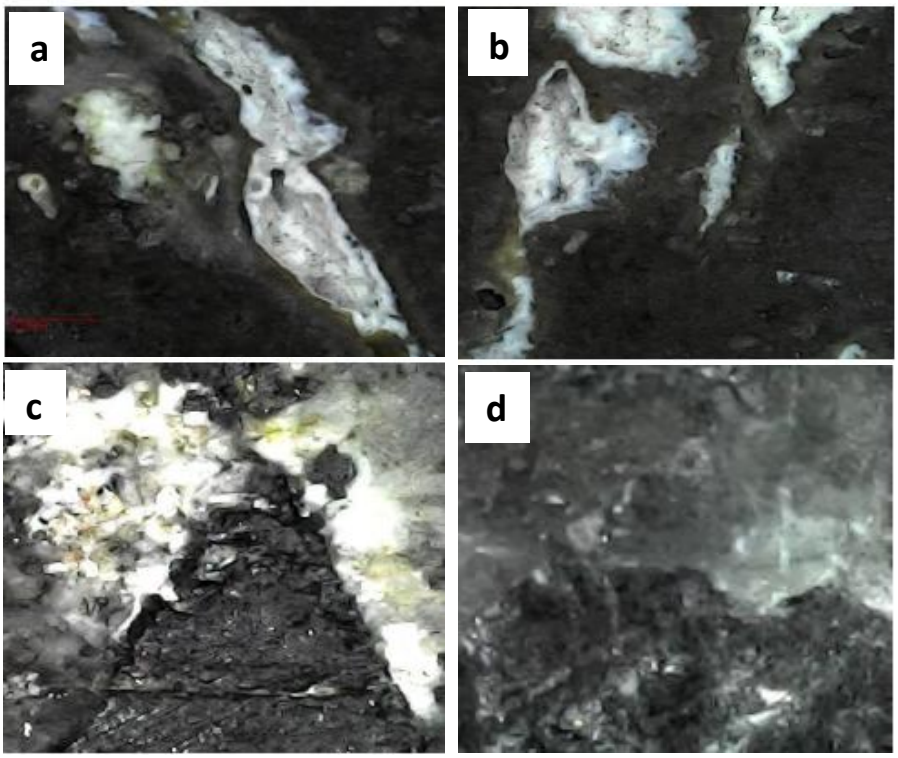

Figure 1. Optical images of all sample A (a and b) and Sample B (c and d)

Quantitative measurements using ImageJ yielded average grain sizes in the range of 20–100 μm, with larger quartz grains contrasted by smaller feldspathic inclusions. This heterogeneous texture is similar to the microfabric observed in karst-related red soils and terra rossa, where micritic calcite and coarser detrital silicates intermingle due to weathering and sedimentation dynamics [7].

Such microtextural observations bridge the chemical findings and the crystalline phase distribution, confirming the coexistence of multiple mineral phases within a structurally complex matrix.

3.3 XRD Analysis

XRD analysis identified the following crystalline phases: calcite ($CaCO_3$)**,** quartz (SiO_2), with minor signatures of albite or related feldspar phases. Sample A showed stronger feldspathic peaks, whereas Sample B exhibited more intense quartz reflections, consistent with its higher Si content. The observed phase assemblages parallel those reported in multi-technique investigations of karst-

derived lithologies elsewhere, such as in multilithic terra rossa deposits from Italy, where XRD confirmed coexistence of calcite and silicate minerals amid weathering residues [7].

Using peak shape analysis and the Debye–Scherrer equation, estimated crystallite sizes ranged between 40–60 nm, indicating moderately nano-crystalline to microcrystalline domains. The crystallinity index, calculated as the ratio of sharp peak area to total diffracted signal (including background), was moderate, implying that amorphous or poorly crystalline phases may be present along with the well-formed mineral grains.

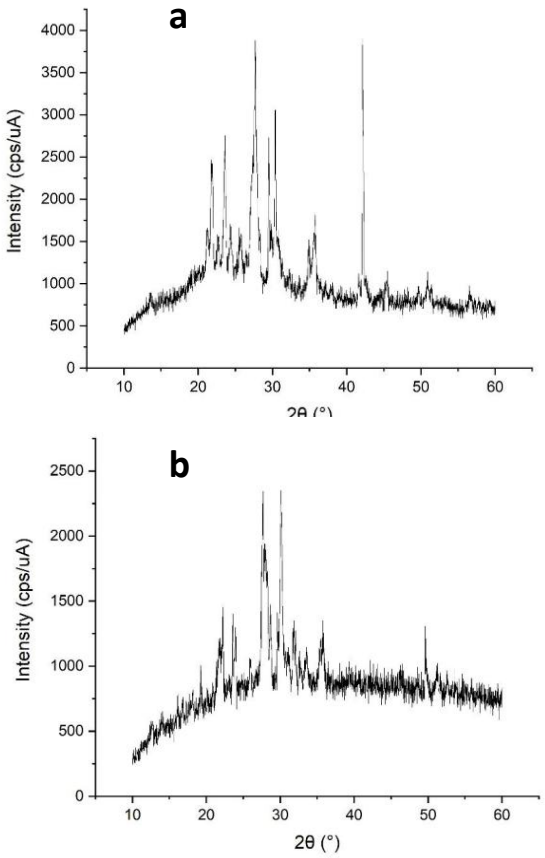

Figure 2. XRD spectrum of a. Sample A, and B. Sample B.

These XRD-based insights confirm the lithological complexity and support interpretations derived from both XRF and optical data.

By integrating XRF, optical microscopy, and XRD data, the study reveals that the rock samples from Leang Panning Park are composed primarily of calcite with significant quartz and feldspathic inclusions—consistent with mixed lithologies typical of karstic environments. The presence of multiple mineral phases, along with varied crystallinity and grain textures, illustrates the interplay between primary geological formation and post-depositional processes such as weathering and diagenesis in karst landscapes [8].

4. Conclusion

The integrated characterization of rock samples from Leang Panning Park, Maros, South Sulawesi using XRF, optical microscopy, and XRD revealed a mixed lithology dominated by calcite and quartz, with minor feldspar contributions. XRF confirmed significant concentrations of Ca and Si, consistent with carbonate and silicate mineral phases, while optical microscopy showed interlocking granular textures with heterogeneous grain sizes. XRD analysis further validated the crystalline phases and indicated moderate crystallinity with nanometer-scale crystallite sizes. Collectively, these findings highlight the geological complexity of the Maros–Pangkep karst region and provide valuable baseline data for future research on mineralogical resources and their potential applications in construction materials, ceramics, and environmental studies.

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